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Trialkylaluminums: Efficient cocatalysts for bis(phenoxy-imine)zirconium complexes in ethylene polymerization

Dongbing Liu [a,*], Shibo Wang [a,b], Hongtao Wang [a], Wei Chen [a]

[a] *Polyolefins National Engineering and Research Center, Sinopec Beijing Research Institute of Chemical Industry, Beijing 100013, PR China*
[b] *Science College, Beijing University of Chemical Technology, Beijing 100029, PR China*

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Abstract

Trialkylaluminums as cocatalysts for bis(phenoxy-imine)Zr complexes, bis[*N*-(3-*tert*-butylsalicylidene)-anilinato]zirconium dichloride (**1**) and bis[*N*-(3-*tert*-butylsalicylidene)-cyclohexylaminato]zirconium dichloride (**2**), are investigated for ethylene polymerization without MAO or $Ph_3CB(C_6F_5)_4$. The effects of alkylaluminums, Al/Zr molar ratio, polymerization temperature and time as well as the comonomer 1-hexene on polymerization performance are examined. In appropriate range of Al/Zr molar ratio, the catalyst system bis(phenoxy-imine)Zr complex/trialkylaluminum shows high activity for ethylene polymerization, which approximates to bis(phenoxy-imine)Zr complex/MAO system. Their catalytic activities depend on not only the nature, but also the amount, of the trialkylaluminum. Both the molecular weights and the polydispersities (M_w/M_n) of PEs produced by **1** with Et_3Al, He_3Al, or Me_3Al are higher than those with MAO. The nature, rather than the amount, of alkylaluminum determines the M_w and the polydispersity of polymer in polymerization with the same complex. Unexpectedly, M_w of the polyethylenes produced with complex **1**/triethylaluminum increases with polymerization time. The catalyst system bis(phenoxy-imine)Zr complex/trialkylaluminum has low capacity of copolymerization.

Keywords: Phenoxy-imine ligand; Zr complex; Trialkylaluminum; Ethylene polymerization; Cocatalyst

1. Introduction

Over the past decade, non-metallocene catalyst systems for olefins polymerization were developing rapidly owing to the diversification and performance of their ligands, as reviewed by Gibson [1,2]. The ligands in precatalysts play the most important roles in olefin polymerization, so do phenoxy-imine ligands, which were first utilized in a zirconium catalyst in 1995 [3]. Grubbs then synthesized a nickel complex with such a ligand for ethylene polymerization [4]. Furthermore, Fujita and his coworkers have successfully established highly active Ti/Zr catalysts using various substituted phenoxy-imine ligands for olefin polymerization, which were named FI catalysts [5–13]. Now phenoxy-imine ligands greatly interest more and more researchers [14,15]. Fluorine-containing phenoxy-imine Ti complex can also be used in living polymerization of propylene as well as ethylene [16–20].

However, the cocatalysts for bis(phenoxy-imine)Zr and Ti complexes are generally MAO (methylaluminoxane), *i*-$Bu_3Al/Ph_3CB(C_6F_5)_4$ [9,21,22], or $MgCl_2$-based activators [23,24]. To the best of our knowledge, there has been no report on common alkylaluminums alone as cocatalyst in the catalytic system of bis(phenoxy-imine)Zr and Ti complexes. Recently we found some alkylaluminums, such as triethylaluminum (Et_3Al), trihexylaluminum (He_3Al), or trimethylaluminum (Me_3Al), could activate bis(phenoxy-imine)Zr and Ti complexes for ethylene polymerization without MAO or $Ph_3CB(C_6F_5)_4$. The activity of bis(phenoxy-imine)Zr complex/trialkylaluminum approaches that of bis(phenoxy-imine)Zr complex/MAO. We now discuss these results in detail.

2. Experimental part

2.1. Materials

All syntheses and manipulations of air-sensitive materials were carried out under a nitrogen atmosphere using Schlenk

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[*] Corresponding author. Fax: +86 10 64273136.
E-mail address: dbliu@brici.ac.cn (D. Liu).

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techniques or in a conventional nitrogen-filled glovebox. Et_3Al, He_3Al, Me_3Al, i-Bu_3Al, and Et_2AlCl were purchased from Crompton Corp., and diluted into their 1.0 M solution in hexane. MAO was purchased from Crompton Corp., and used as received. Toluene was refluxed over sodium/benzophenone ketyl and distilled under nitrogen before use. Hexane was dried over 4 Å molecular sieves. Ethylene was obtained from the Yanshan Petrochemical Corporation, China Petroleum & Chemical Corporation.

2.2. Analysis methods

^1H NMR spectra were recorded on a Bruker DMX 400 spectrometer (400 MHz) in $CDCl_3$. Elemental analysis for C, H, and N was performed on an EA1112 instrument from CE. Mass spectra CI-MS were recorded on a QUATTRO II instrument from Macro Mass. Weight-average molecular weights (M_w) and molecular weight distributions (M_w/M_n) of polymers were measured by means of a Waters gel permeation chromatograph Alliance GPCV 2000 at 150 °C using 1,2,4-trichlorobenzene as eluent. ^{13}C NMR spectra of PEs were recorded with a Bruker DMX 400 spectrometer operating at 100.6 MHz, on 10–20 mg/mL solutions in deuterated dichlorobenzene at 120 °C. Melting points of polymers were obtained on a Perkin-Elmer DSC-7 by heating from 0 to 160 °C at a heating rate of 10 °C/min.

2.3. Complex synthesis

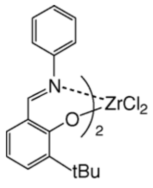
Complexes **1** and **2** were synthesized by the reaction of $ZrCl_4(THF)_2$ with two equivalents of lithium salt of the corresponding phenoxy-imine ligands according to the literature [8].

2.3.1. Bis[N-(3-tert-butylsalicylidene)-anilinato]zirconium(IV) dichloride (*1*)

^1H NMR ($CDCl_3$, δ): 1.33–1.59 ppm (m, 18H, *t*Bu), 6.79–7.42 ppm (m, 16H, aromatic-H), 8.12 ppm (s, 2H, CH=N). $C_{34}H_{36}Cl_2N_2O_2Zr$: Calcd. C 61.26%, H 5.41%, N 4.20%; Found C 61.29%, H 5.75%, N 4.68%. CI-MS: m/z 665 $[(M+1)^+]$.

2.3.2. Bis[N-(3-tert-butylsalicylidene)-cyclohexylaminato]zirconium(IV) dichloride (*2*)

^1H NMR ($CDCl_3$, δ): 1.60 ppm (s, 18H, *t*Bu), 1.15–2.10 ppm (m, 20H, CH_2), 6.90–7.58 ppm (m, 6H, aromatic-H), 8.23 ppm (s, 2H, CH=N). $C_{34}H_{48}Cl_2N_2O_2Zr$: Calcd. C 60.18%, H 7.08%, N 4.13%; Found C 60.34%, H 7.24%, N 4.31%. CI-MS: m/z 678 $[M^+]$.



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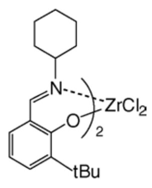

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2.4. Polymerization procedure

The ethylene polymerization was carried out in a jacketed 1 L glass reactor (Büch Corporation) connected to a 5 L oil-bath (Huber Corporation). The reactor was purged with nitrogen and ethylene for three times, respectively. Then 500 mL dry *n*-hexane was added into the reactor with stirring. At the same time, the hexane solution of AlR_3 was injected followed by a prescribed amount of the catalyst solution in toluene at room temperature. In the polymerization temperature and the pressure of ethylene were maintained. After 30 min, the polymerization was terminated, the resulted polymer was filtered and dried at room temperature until constant weight was attained.

3. Results and discussion

3.1. Alkylaluminum cocatalyst

Alkylaluminums were investigated as cocatalysts for complexes **1** and **2** in catalytic polymerization of ethylene. The results are listed in Table 1. It is found that only the alkylaluminum with three linear alkyls can activate complexes **1** and **2**. Comparing Et_3Al, He_3Al, Me_3Al with MAO (see Table 1 entries 1–8 and 13), the activity of **1** with Et_3Al is similar to that of **1** with MAO at the same Al/Zr molar ratio, whereas the activity of **1** with He_3Al or Me_3Al is lower than with MAO. Activation efficiency of trialkylaluminums to complexes **1** and **2** is different: the activity of **1** is decreasing in the order $Et_3Al > Me_3Al > He_3Al$, while the order for **2** is $Et_3Al > He_3Al > Me_3Al$. It has been reported that i-Bu_3Al and Et_2AlCl are effective cocatalysts for late transition metal catalysts in polymerization of ethylene [25–27]. However, no polymer was found using i-Bu_3Al or Et_2AlCl as cocatalyst for complex **1**.

The activities of **1** and **2** obviously depend on both the kind and the amount of trialkylaluminum. For example, complex **1**/Et_3Al reached the maximum activity (6.16×10^7 g/mol Zr h) with the Al/Zr molar ratio of 15,000 (Table 1 entries 1–4), while complex **2** and Et_3Al showed the maximum activity (57.8×10^6 g/mol Zr h) with the Al/Zr molar ratio of 10,000.

The molecular weights of the polyethylenes produced by **1** with Et_3Al, He_3Al, or Me_3Al are much higher than those produced with MAO. Also the polydispersities (M_w/M_n) of polyethylenes produced by **1** with trialkylaluminums are higher than with MAO.

According to Table 1, the weight-average molecular weights (M_w) and the polydispersities (M_w/M_n) of PEs with the same complex are chiefly relied on the nature of the alkylaluminum, and the amount of the alkylaluminum has less effect on M_w and M_w/M_n. This is also shown by the GPC curves of PEs prepared with complex **2** in Fig. 1. It is conceivable that the active species are relative to different alkylaluminums and the chain transfer to aluminum is not the main chain-transfer pathway.

Like most of the single site catalysts, the structure of ligands in this catalyst system has an effect on the molecular weight and polydispersity of polyethylene. With the same alkylaluminum as

Table 1
Ethylene polymerization results with complexes **1** and **2** activated by alkylaluminums

Entry[a]	Complex	Cocatalyst	Al/Zr	Yield (g)	Activity[b]	M_w ($\times 10^{-4}$)	M_w/M_n
1	**1**	Et_3Al	4000	6.30	25.2	26.0	69.4
2	**1**	Et_3Al	10000	10.85	43.4	22.6	43.6
3	**1**	Et_3Al	15000	15.40	61.6	30.0	71.6
4	**1**	Et_3Al	20000	10.71	42.8	22.6	42.7
5	**1**	He_3Al	4000	1.64	6.56	20.2	31.2
6	**1**	He_3Al	10000	0.34	1.36	20.3	28.6
7	**1**	Me_3Al	4000	2.87	11.5	9.1	16.7
8	**1**	Me_3Al	10000	2.96	11.8	12.6	21.5
9	**1**	i-Bu_3Al	4000	0			
10	**1**	i-Bu_3Al	10000	0			
11	**1**	Et_2AlCl	4000	0			
12	**1**	Et_2AlCl	10000	0			
13	**1**	MAO	10000	12.10	48.4	1.2	2.40
14	**2**	Et_3Al	4000	8.24	33.0	7.7	10.6
15	**2**	Et_3Al	10000	14.46	57.8	7.5	7.34
16	**2**	Et_3Al	20000	10.20	40.8	8.0	6.33
17	**2**	He_3Al	4000	7.14	28.6	4.7	3.36
18	**2**	He_3Al	10000	7.86	31.4	4.5	3.28
19	**2**	He_3Al	20000	2.65	10.6	5.1	3.19
20	**2**	Me_3Al	4000	2.18	8.72	7.1	7.36
21	**2**	Me_3Al	10000	0.67	2.68	7.9	9.28

[a] Conditions: 0.5 μmol complex, 500 mL hexane, 0.1 MPa ethylene, polymerization temperature = 30 °C, polymerization time = 30 min.
[b] In 10^6 g/mol Zr h.

cocatalyst, the polyethylenes produced by complex **1** are wider in polydispersity than those by complex **2**.

It has been reported that the reaction of Schiff base complex or Schiff base ligand with trimethylaluminum is capable of producing phenoxy-imine aluminum complex or species, and complex [*N*-(3-*tert*-butylsalicylidene)-anilinato]-dimethylaluminum has extra low activity (400 g/mol h atm) in ethylene polymerization [28–30]. Thus, it could be confirmed that the metal center of the active species in the catalytic system of bis(phenoxy-imine)Zr complex/trialkylaluminum still is zirconium, not aluminum, and trialkylaluminum acts as the cocatalyst during ethylene polymerization.

Fujita has reported that bis(phenoxy-imine)zirconium complexes potentially possess five isomers which might result in multiple active species under appropriate conditions [31]. By changing substitutes of the ligands and varying polymerization temperature, the catalyst system bis(phenoxy-imine)zirconium/MAO produced polyethylenes with multi-modal MWDs. However, the complex **1**/MAO system produced polyethylenes with single modal MWDs in the report.

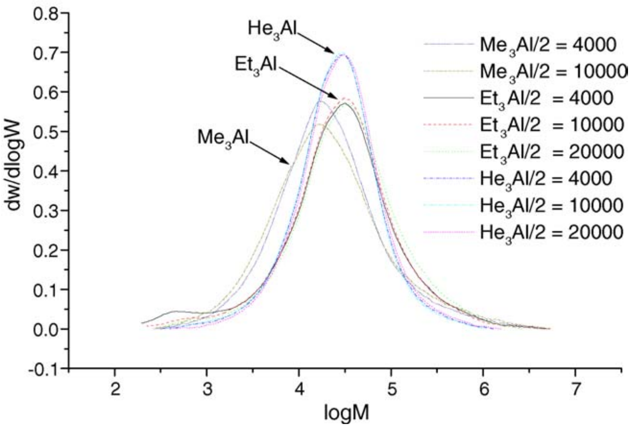

Fig. 1. GPC curves of PEs prepared with catalyst systems **2**/Et_3Al, **2**/He_3Al and **2**/Me_3Al.

From the above discussion, it is speculated that the broad MWDs of PEs are probably caused by multiple active species existing in the new catalyst system, bis(phenoxy-imine)zirconium/alkylaluminum.

3.2. Polymerization temperature

Table 2 lists the results of ethylene polymerization with complexes **1** and **2** activated by triethylaluminum at different temperatures. At 30 °C, catalytic activities are highest in both systems of complexes **1** and **2** with triethylaluminum. Their catalytic activities are decreased at either higher or lower polymerization temperature. However, in the catalyst system complex **1**/MAO, the optimum polymerization temperature is 50 °C [8]. The possible reason is that trialkylaluminum may destroy the complex at high temperature, and cause the catalyst to deactivate. In our another research, we have found that the supported catalyst containing complex **1** or **2** still has a high activity at 70–80 °C activated by trialkylaluminum.

With increasing polymerization temperature, molecular weights and M_w/M_n change in no orderliness in catalyst system complex **1**/$AlEt_3$, while the M_ws and M_w/M_n decrease in catalyst system complex **2**/$AlEt_3$. This is clearly shown by GPC curves of polymers (Fig. 2), and two peaks appeared in the GPC curve of polymer produced at 50 °C.

Table 2
Results of ethylene polymerization with complexes **1** and **2**/triethylaluminum under different polymerization temperature

Entry[a]	Complex	Polymerization temperatrure (°C)	Yield (g)	Activity[b]	M_w ($\times10^{-4}$)	M_w/M_n
1	**1**	10	1.61	6.44	10.3	21.1
2	**1**	30	6.30	25.2	26.0	69.4
3	**1**	50	1.05	4.20	18.1	46.5
4	**2**	10	3.84	15.4	15.6	17.3
5	**2**	30	8.24	33.0	7.7	10.6
6	**2**	50	4.72	18.9	3.0	7.67

[a] Conditions: 0.5 μmol complex, 2 mmol Et_3Al, 500 mL hexane, 0.1 MPa ethylene, polymerization time = 30 min.
[b] In 10^6 g/mol Zr h.

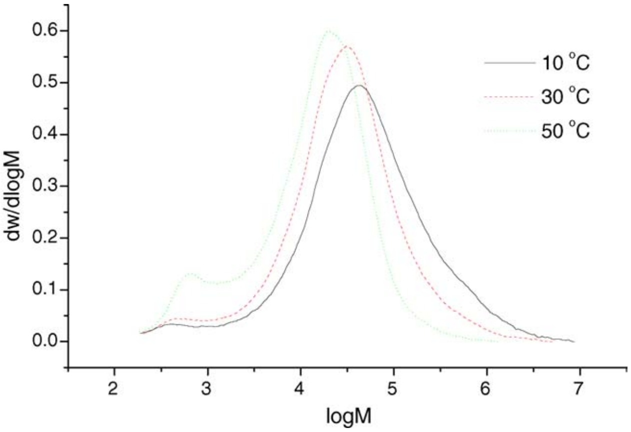

Fig. 2. GPC curves of PEs prepared with catalyst system **2**/Et_3Al at different polymerization temperature.

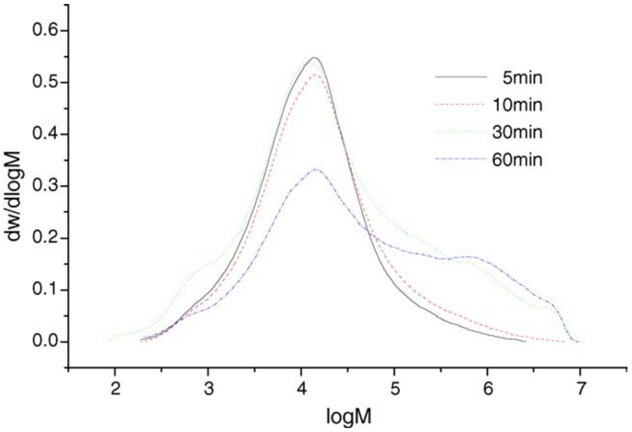

Fig. 3. GPC curves of PE prepared with catalyst system **1**/Et_3Al at different polymerization time.

3.3. Polymerization time

The polymerization results with complex **1**, activated by triethylaluminum with different polymerization time, are listed in Table 3. As polymerization time increases, the catalytic activity decreases quickly. Interestingly the M_w of polyethylene produced with complex **1**/triethylaluminum increases with polymerization time. For example, the M_w of polyethylene made in 5 min is 42,000 g/mol while M_w of polyethylene made in 60 min was 419,000 g/mol.

The M_w/M_n value of PEs produced with complex **1**/triethylaluminum becomes higher along with increasing polymerization time. From the GPC curves of polymers in Fig. 3, it is observed that polyethylene produced within 60 min is multi-modal, and the peak position of the low molecular weight range obtained in 60 min is the same as the peak position of the polymer produced within 5 min. Prolonging polymerization time, the weight of polymer with high molecular weight increased.

Considering the chain termination mechanism, β-H transfer produces the vinyl end group and chain transfer to aluminum produces methyl end group. In view of the conventional single site catalyst, the content of saturated methyl end-group decreases with increasing polymerization time due to depletion of alkylaluminum. But the content of methyl end group in Table 3 increases with polymerization time. This result also reveals that there may be multiple active species that have different polymerization performances in catalyst system bis(phenoxy-imine)Zr complex/trialkylaluminum.

The GPC curves for PEs produced with complex **1**/triethylaluminum in 5 and 60 min are resolved into several components in Gauss fit with mathematical calculations software Microcal Origin® (see Fig. 4). Each component has a value of

Table 3
Results of ethylene polymerization with complex **1**/triethylaluminum at different polymerization time

Entry[a]	Polymerization time (min)	Yield (g)	Activity[b]	M_w ($\times10^{-4}$)	M_w/M_n	Chain-end group[c] vinyl/methyl
1	5	2.83	67.9	4.2	8.26	36/64
2	10	4.08	49.0	7.2	12.76	–
3	30	6.30	25.2	26.0	69.42	–
4	60	9.18	18.4	41.9	60.38	23/77

[a] Conditions: 0.5 μmol complex **1**, 2 mmol Et_3Al, 500 mL hexane, 0.1 MPa ethylene, polymerization temperature = 30 °C.
[b] In 10^6 g/mol Zr h.
[c] In mol% by ^{13}C NMR.

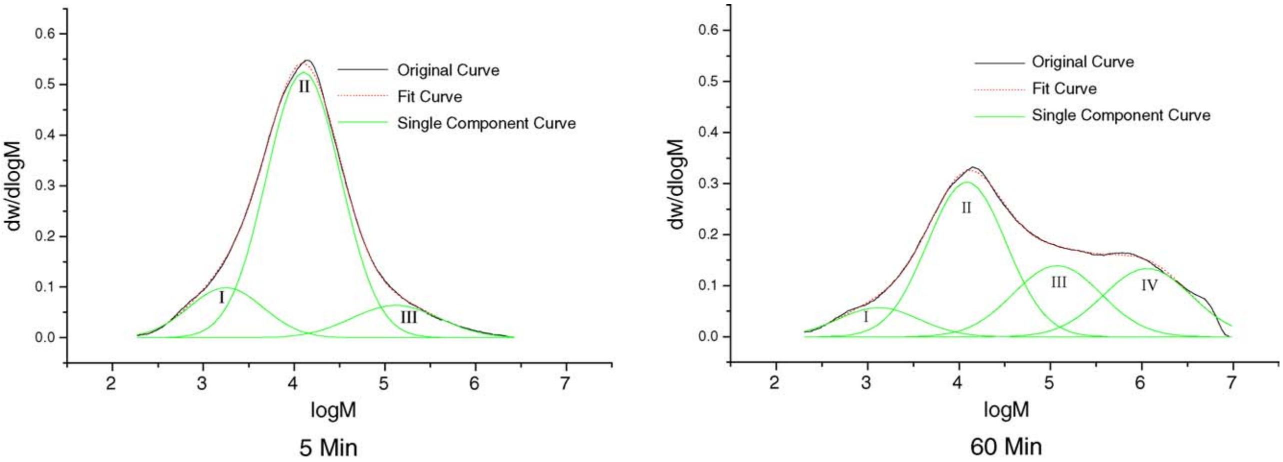

Fig. 4. GPC curves and their resolution into Gauss components for PEs prepared with catalyst system **1**/Et_3Al at different polymerization time.

Table 4
The value of each component in Fig. 4

Time (min)	M_w (av.)	M_w/M_n	Component	M_w	M_w/M_n	Fraction (%)
5	42000	8.26	I	2777	2.31	14.2
			II	19770	2.43	75.1
			III	230218	3.25	10.7
60	419000	60.38	I	2248	2.41	4.6
			II	19864	2.67	45.6
			III	217104	3.38	23.4
			IV	1762658	2.91	22.4

Table 5
Results of ethylene/1-hexene copolymerization with complex **2**/trihexylaluminum

Entry[a]	1-Hexene (mL)	Yield (g)	Activity[b]	M_w ($\times 10^{-4}$)	M_w/M_n	Content of 1-henxene[c]	T_m × °C
1	0	7.14	28.6	4.7	3.36	0.00	132.9
2	10	4.70	18.8	4.0	3.11	0.05	132.4
3	30	1.29	5.16	3.6	2.93	0.16	131.0

[a] Conditions: 0.5 μmol complex **2**, 2 mmol He_3Al, 500 mL hexane, 0.1 MPa ethylene, polymerization temperature = 30 °C, polymerization time = 30 min.
[b] In 10^6 g/mol Zr h.
[c] In mol% by ^{13}C NMR.

M_w/M_n 2.2–3.4, which is similarly regarded as the performance of single site catalyst. Fig. 4 shows that the new component IV with higher molecular weight appears in GPC curve of polyethylene produced in 60 min. The data listed in Table 4 indicates that contents of components I and II are decreasing while contents of component III and IV are increasing due to change of reaction time from 5 to 60 min. It is therefore suggested that there are multiple active species that have different kinetics in catalyst system bis(phenoxy-imine)Zr complex/trialkylaluminum.

3.4. Effect of comonomer

Copolymerization was studied with complex **2**/trihexylaluminum by adding 1-hexene into the reactor. The results listed in Table 5 show that the activity decreases greatly as increasing the volume of 1-hexene. In addition, the weight average molecular weight and M_w/M_n decrease slightly. Less content of 1-hexene and less decrease in T_ms reveal that catalyst system **2**/He_3Al has low copolymerization capability.

4. Conclusion

In conclusion, common trialkylaluminums such as triethylaluminum (Et_3Al), trihexylaluminum (He_3Al) and trimethylaluminum (Me_3Al) are efficient cocatalysts to activate bis(phenoxy-imine)Zr complexes for ethylene polymerization without MAO or $Ph_3CB(C_6F_5)_4$. In the catalyst system bis(phenoxy-imine)zirconium/trialkylaluminum, there probably exist multiple active species, which are relative with alkylaluminum.

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